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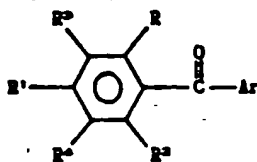
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(84) Substituted benzophenones and their liquid mixtures suitable for use as photopolymerisation initiators.

(87) As initiators for the photopolymerisation of ethylenically unsaturated compounds, the use of systems consisting of compounds of benzophenone type of formula I



In which R, R<sup>1</sup> and R<sup>2</sup> are preferably alkyls of 1-4 carbon atoms, R<sup>3</sup> and R<sup>4</sup> are preferably hydrogen and Ar is preferably phenyl, in combination with donors of hydrogen or substances which determine the formation of labile photochemical complexes, such as alcohols, ethers and tertiary amines with hydrogens in the alpha position, and the use for the same purpose of mixtures of 2,4,6-trialkylbenzophenones and benzophenone and particularly of a mixture of 2,4,6-trimethylbenzophenone and benzophenone in a molar ratio of 1.155, which has the advantage of being liquid at temperatures exceeding 10°C and of possessing photochemical activity superior to that of benzophenone.

EP 0 209 831 A2

- 1 -

SUBSTITUTED BENZOPHENONES AND THEIR LIQUID MIXTURES SUITABLE FOR  
USE AS PHOTOPOLYMERISATION INITIATORS

This invention relates to the use of systems comprising  
5 substituted benzophenones and their liquid mixtures as photopoly-  
merisation initiators for ethylenically unsaturated compounds.

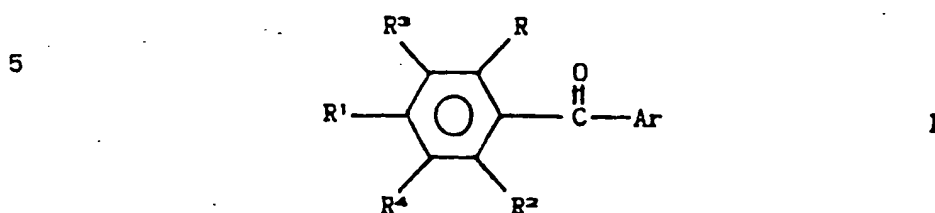
Ethylenically unsaturated compounds and in particular acrylic and  
methacrylic acid derivatives can be polymerised by irradiation  
10 with ultraviolet light of wavelength between 200 and 450 nm in  
the presence of photoinitiator systems formed from:

- A) benzophenone or its derivatives
- B) a donor of hydrogen or substances which determine the  
formation of a labile photochemical complex with the carbonyl  
15 compound (A), such as alcohols, tertiary amines or ethers having  
available hydrogens on the carbon adjacent to the heteroatom.

Examples of benzophenones which form these photoinitiator systems  
are benzophenone itself which is the most widely used, 4,4'-bis-  
20 dimethyl(or diethyl) aminobenzophenone (Michler ketone), which is  
very efficient in pigmented systems, 4-hydroxybenzophenone  
condensed with ethylene oxide and esterified with acrylic acid,  
which has the advantage of being liquid and copolymerisable, 2-  
carbomethoxybenzophenone, 3,3',4,4'-benzophenonetetracarboxylic  
25 acid esters etc. As many of these products are solid, they are  
difficult to incorporate into the photopolymerisable systems, or  
have a photochemical efficiency which is less than benzophenone  
itself.

In accordance with the present invention, we have now surprisingly discovered that systems consisting of:

A) compounds of formula I



in which

- 10 R signifies linear or branched C<sub>1</sub>-C<sub>12</sub> alkyl possibly substituted with one or more phenyl, chlorine, bromine, C<sub>1</sub>-C<sub>12</sub> alkoxy, ArO-, ArSO-, ArSO<sub>2</sub>-, C<sub>1</sub>-C<sub>12</sub> alkylsulphinyl or C<sub>1</sub>-C<sub>12</sub> alkylsulphonyl groups; or -SO<sub>3</sub>H or C<sub>1</sub>-C<sub>12</sub> dialkylamino, respectively as free acids or bases or
- 15 salified with organic or inorganic bases; C<sub>1</sub>-C<sub>12</sub> alkoxy; or ArO-
- R¹, R² which can be equal or different, each independently have the same meaning as R or represent hydrogen or a bridge of carbon atoms with Ar or R³ or R⁴ possibly comprising
- 20 heteroatoms
- R³, R⁴ which can be equal or different, each independently represent hydrogen, linear or branched C<sub>1</sub>-C<sub>12</sub> alkyl, phenyl, chlorine, bromine, -SO<sub>3</sub>H possibly salified with organic or inorganic bases, or ArCO-
- 25 Ar represents a C<sub>6</sub>-C<sub>10</sub> aryl either non-substituted or carrying one or more substituents of the halogen, C<sub>1</sub>-C<sub>12</sub> alkyl, phenyl, C<sub>1</sub>-C<sub>4</sub> alkoxy, C<sub>3</sub>-C<sub>6</sub> cycloalkoxy, phenoxy, -COOH, C<sub>1</sub>-C<sub>6</sub> alkoxycarbonyl, benzoyl, C<sub>1</sub>-C<sub>6</sub> dialkylamino, C<sub>1</sub>-C<sub>6</sub> alkylthio, alkylsulphinyl, alkylsulphonyl,
- 30 arylthio, arylsulphinyl and arylsulphonyl group; or 2,3 or 4-pyridyl; or a furan or thiophene residue
- B) an organic tertiary amine of aliphatic type, or an ester of p-dimethylaninobenzoic acid or 4,4'-bis-(C<sub>1</sub>-C<sub>6</sub> dialkylamino) benzophenone, or single or polymeric ethers of primary or
- 35 secondary alcohols or glycols or primary or secondary aliphatic alcohols or in any event donors of hydrogen or substances which determine the formation of labile chemical complexes,

can be used in the photoinitiated polymerisation of ethylenically unsaturated compounds, and in particular of acrylic and methacrylic acid derivatives, with a photochemical efficiency considerably greater than that of analogous systems containing the currently used benzophenones indicated heretofore.

Further according to the invention, we have also surprisingly found that particular mixtures of benzophenone and compounds of formula I, by virtue of having melting points considerably lower than those of the individual compounds, can be used in the liquid state with considerable practical advantages with regard to their incorporation into the photopolymerisable mixtures, in combination with the compounds of type B indicated heretofore.

Preferred systems for use according to the present invention are those consisting of:

- A) one or more benzophenones of formula I in which  
R, R', R<sup>2</sup> which can be equal or different, each independently signify C<sub>1</sub>-C<sub>4</sub> alkyl,
- 20 R<sup>3</sup>, R<sup>4</sup> which can be equal or different, each independently signify hydrogen, C<sub>1</sub>-C<sub>4</sub> alkyl, or -SO<sub>3</sub>H possibly salfified with organic or inorganic bases,
- Ar signifies phenyl, possibly substituted with one or more C<sub>1</sub>-C<sub>4</sub> alkyl, Cl, Br or C<sub>1</sub>-C<sub>4</sub> alkylthio groups
- 25 B) an organic tertiary amine of aliphatic type, or an ester of p-dimethylaminobenzoic acid or 4,4'-bis-(C<sub>1</sub>-C<sub>4</sub> dialkylamino) benzophenone, or molecules containing single or polymeric derivatives of glycols.

30 Examples of compounds of formula 1 are:

- 2,4,6-trimethylbenzophenone
- 2,4,6-triethylbenzophenone
- 2,4,6-triisopropylbenzophenone
- 2,4,6-tritert. butylbenzophenone
- 35 2,4,6-trimethoxybenzophenone
- 2,3,4,5,6-pentamethylbenzophenone
- sodium 2,4,6-trimethylbenzophenone-3-sulphonate

4-(2,4,6-trimethylbenzoyl)pyridine

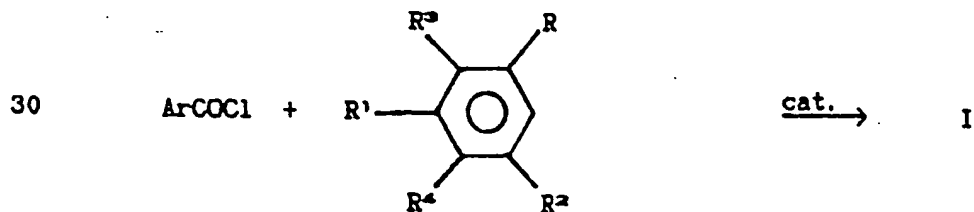
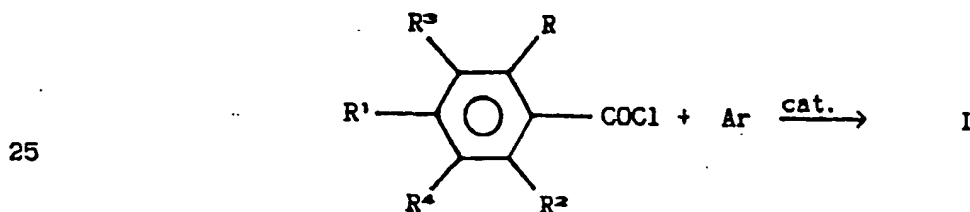
2-(2,4,6-trimethylbenzoyl)pyridine.

Preferred as liquid mixtures of compounds of formula I and  
5 benzophenone are those with molar ratios of between 0.1 and 10.

Particularly preferred as liquid mixtures of compounds of formula  
I and benzophenone are those of 2,4,6-trimethylbenzophenone and  
benzophenone in molar ratios of between 1 and 1.3 having a pour  
10 point less than 0°C.

Particularly preferred compounds of type B are single polymeric  
derivatives of aliphatic alkanolamines, esters of p-dialkyl-  
aminobenzoic acid, and aliphatic alcohols and glycols possibly  
15 carrying acrylic saturations or included in macromolecular  
structures.

Compounds of formula I are known from the scientific literature  
and are normally obtained by Friedel-Crafts acylation (Houben  
20 Veyl, Methoden der Organischen Chemie, Band VII/2a, Teil 1, pag.  
164-233) in accordance with the two following alternatives:



where cat. signifies a Friedel-Crafts catalyst. For example  
2,4,6-trimethylbenzophenone has been obtained by acylation of  
35 mesitylene with benzoyl chloride in the presence of anhydrous  
aluminium chloride [Louise, Annales del Chemie, 6 (6) 202; H.O.  
House et al., J. Org. Chem., 41, 3083 (1976)].

Although there are numerous publications in the literature on the photochemistry and photophysics of the compounds of formula I, and in particular of 2,4,6-trialkylbenzophenones [Y. Kitaura et al., Tetrahedron, 27 1597 (1971) - Y. Ito et al., J. Am. Chem. Soc. 102, 5917 (1980), ibid. 105, 1590 (1983), J. Org. Chem., 46, 4359 (1981) - G. Porter et al., J. Chem. Soc. (A) 3772 (1971), Chem. Comm., 1372 (1970)] there is no reference to their use as polymerisation photoinitiators.

10 The photopolymerisable compounds with which the photoinitiator systems according to the present invention can be advantageously used are di, tri, tetra or polyfunctional monomers and oligomers of acrylic or methacrylic type, such as esters or amides.

15 Photopolymerisable oligomers and prepolymers are represented most commonly by polyester, polyether, polyurethane, acrylic, epoxy and silicone resins containing acrylic or methacrylic functionalities. Those mixtures useful in the art are those formed from the aforesaid monomers and oligomers in possible  
20 combination with pigments, fillers, thermal stabilisers, light stabilisers, antioxidants, paraffins etc., various auxiliaries such as anti-foaming agents, dispersants etc. The co-use of other photoinitiators such as benzyl ketals, benzoin ethers, thioxanthenes etc. is also possible.

25 Photopolymerisable mixtures containing the photoinitiator systems according to the present invention can be used as coatings for wood, metal, paper, fabrics, plastics materials, fibreglass, rubber; printing inks, adhesives and sealants; masses for  
30 fabricating printing plates or silk-screen matrices; masses for fabricating structural materials.

With regard to light sources, the photopolymerisation process can use medium, low or high pressure mercury vapour lamps, super-  
35 actinic lamps or lamps specially designed to obtain high radiation intensity at wavelengths between 250 and 450 nm.

The photoinitiator systems according to the present invention are used in a quantity of between 0.1 and 15% by weight of the photopolymerisable mixture, and preferably between 0.5 and 10% by weight. The ratio of component A to component B can vary between  
5 0.1 and 10 but values of between 0.5 and 1.5 are preferred.

When the compounds of formula I are used as the benzophenone system of type A, three advantages are obtained over the use of benzophenone itself, namely better photochemical efficiency and  
10 less film coloration and odour.

When liquid mixtures of compounds of formula I with benzophenone are used, in addition to the stated advantages the preparation of photopolymerisable mixtures takes less mixing time and lower  
15 temperatures can be used. In addition the fact that a liquid photoinitiator is used helps to keep the viscosity low and allows co-solubilisation of components which would otherwise be more difficult to dissolve.

20 The following examples illustrate the invention in greater detail, but without limiting it (the parts are by weight).

The benzophenones used are indicated by abbreviations and have the following characteristics:

- 25 BF = benzophenone - crystalline solid, M.P. 49°C, IR spectrum (KBr) : 1655, 1275, 695, 633  $\text{cm}^{-1}$ .
- TMB = 2,4,6-trimethylbenzophenone - metastable liquid, B.P. 189°C (17 mmHg) or crystalline solid with M.P. 35.5°C, IR spectrum (KBr) : 1670, 1270, 917, 710  $\text{cm}^{-1}$ .
- 30 TEB = 2,4,6-triethylbenzophenone - liquid, B.P. 210-220°C (40 mmHg), IR spectrum (liquid film) : 1660, 1270, 925, 865, 705  $\text{cm}^{-1}$ .
- TIPD = 2,4,6-triisopropylbenzophenone - crystalline solid, M.P. 98-99°C, IR spectrum (KBr) : 1665, 1250, 950, 930, 880,  
35 725  $\text{cm}^{-1}$ .
- PMB = 2,3,4,5,6-pentamethylbenzophenone - crystalline solid, M.P. 134-135°C, IR spectrum (KBr) : 1670, 1210, 890, 705

cm<sup>-1</sup>.

TMOB = 2,4,6-trimethoxybenzophenone - crystalline solid, M.P. 112-113°C, IR spectrum (KBr) : 1660, 1600, 1585, 1125, 810 cm<sup>-1</sup>.

5 MDEA = N-methyldiethanolamine - liquid.

#### EXAMPLE 1

Eutectic mixture of 2,4,6-trimethylbenzophenone and benzophenone (BF/TMB)

10

585 g of 2,4,6-trimethylbenzophenone (M.P. 35.5°C) are heated to 45°C, 415 g of benzophenone (M.P. 49°C) are added, and after agitation for 15 minutes the mixture is cooled to 20°C. A composition is obtained (molar ratio TMB/BF = 1.155) having the following characteristics; IR spectrum (liquid film) : 1660, 1270, 905, 700 cm<sup>-1</sup>; clear liquid up to 10°C; pour point (ASTM D97/66) less than -10°C; Brookfield viscosity RVT 1/20 rpm = 55 mPas at 20°C,  $\eta$  = 145 mPas at 10°C.

#### 20 EXAMPLES 2 to 5

The photo-crosslinkable mixtures having the composition indicated in the individual examples given hereinafter were applied to a glass plate to a thickness of 50 microns. After exposure to air for 30 seconds the films were irradiated by successive passages at the indicated conveying speed under a medium pressure mercury vapour lamp of the indicated power at a distance of 10 cm from the light source. Sward hardness (ASTM D2134-66) and yellowing (ASTM D1925-63T) were determined.

30 The thermal stability, evaluated for photoinitiated mixtures kept in darkness at 60°C, was greater than 30 days for all the photoinitiator systems.

In Example 2, the operating conditions and results were as follows:

Photo-crosslinkable composition	Laromer <sup>®</sup> LR 8496 (acrylated aliphatic epoxy resin BAST <sup>®</sup> (B))
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95-93



- 8 -

MDEA 1-3

Benzophenone compound (50% solution  
in 1,6-hexanedioldiacrylate) (A) 4

Lamp power 40 W/cm

5 Conveying speed 20 m/min.

	A	MDEA parts by wt	R parts by wt	Film tacki- ness after 1 passage	Sward hardness after			Yellowing Index after 4 passages
					2 passages	3 passages	4 passages	
10	BF (a)	1	95	liquid	6	10	14	5
		2	94	tacky	8	12	14	6
		3	93	slightly tacky	10	10	14	6
	TMB	1	95	tacky	8	12	16	3
		2	94	slightly tacky	10	12	16	3
15		3	93	dry	10	12	16	3
	BF/TMB	1	95	slightly tacky	8	12	14	4
		2	94	dry	8	12	16	4
		3	93	dry	10	12	16	4

(a) BF was used for comparison purposes

20

In Example 3, the operating conditions and results were as follows:

Photo-crosslinkable composition 25 Laromer<sup>®</sup> EA 81 (acrylated aromatic epoxy resin BASF) (R) 86

1,6-hexanediol diacrylate (HDDA) 0-12.25

benzophenone compound (50% solution in HDDA) (B) 1-8

MDEA 0.75-6

Lamp power 40 W/cm

30 Conveying speed 20 m/min.

	B	B parts by wt	MDEA parts by wt	HDDA parts by wt	Sward hardness after				
					1 passage	2 passages	3 passages	4 passages	5 passages
35	BF (a)	1	0.75	12.25	tacky	2	6	6	8
		2	1.5	10.50	12	18	20	24	24
		4	3.0	7.0	12	20	24	26	30

		8	6.0	-	8	16	20	26	28
	TMB	1	0.75	12.25	8	10	12	12	12
		2	1.5	10.50	16	22	22	28	28
		4	3.0	7.0	14	24	28	32	32
5		8	6.0	-	12	22	26	26	30
	BF/TMB	1	0.75	12.25	4	8	10	10	12
		2	1.5	10.50	12	18	22	26	26
		4	3.0	7.0	14	22	28	30	32
		8	6.0	-	12	20	12	26	28
10	PMB	2	1.5	10.50	-	22	22	26	26
	TIPB	2	1.5	10.50	-	18	22	24	26
	TMB	2	1.5	10.50	-	16	20	22	24

(a) BF was used for comparison purposes

15 In Example 4, the operating conditions and results were as follows:

Photo-crosslinkable composition	Laromer <sup>®</sup> EA 81	86
	benzophenone compound (18.2% solution in vinylpyrrolidone) (B)	11
20	MDEA	3
Lamp power	40 W/cm	
Conveying speed	20 m/min.	

B	Sward Hardness after:			
	1 passage	2 passages	3 passages	4 passages
25				
BF (a)	22	30	32	38
TMB	24	32	38	40
TIPB	18	32	38	40
PMB	20	30	36	38
30				
TMB	26	28	34	38

(a) BF was used for comparison purposes

In Example 5, the operating conditions and results were as follows:

35	Photo-crosslinkable composition	Laromer <sup>®</sup> 55 F (acrylated polyester resin BASF)	100
		benzophenone compound (A)	2

- 10 -

Silicone Dow DC 190 (20% solution  
in xylene)

0.2

Film thickness . 500 microns

Lamp power 80 W/cm

5

A Yellowing index of crosslinked film after:

1 passage at 3.5 m/min 10 passages at 1 m/min

BF (a) 2 15

TMB 2 10

10 2,2-dimethoxy-2-

phenylacetophenone (b) 10 20

1-benzoylcyclohexanol (b) 5 12

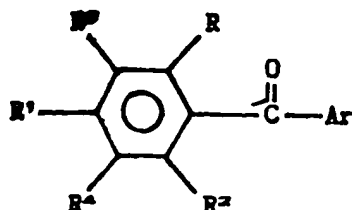
(A) used for comparison purposes

(b) non-benzophenone compounds used for comparison purposes.

## PATENT CLAIMS

1. As initiators for the photopolymerisation of ethylenically unsaturated compounds, the use of systems consisting of:

5 A) one or more benzophenones of formula I



I

10 in which

R signifies linear or branched  $C_1$ - $C_{12}$  alkyl possibly substituted with one or more phenyl, chlorine, bromine,  $C_1$ - $C_{12}$  alkoxy,  $ArO$ -,  $ArSO$ -,  $ArSO_2$ -,  $C_1$ - $C_{12}$  alkylsulphinyl or  $C_1$ - $C_{12}$  alkylsulphonyl groups; or  $-SO_3H$  or  $C_1$ - $C_{12}$  dialkylamino, respectively as free acids or bases or salified with organic or inorganic bases;  $C_1$ - $C_{12}$  alkoxy; or  $ArO$ -

$R^1$ ,  $R^2$  which can be equal or different, each independently have the same meaning as R or represent hydrogen or a bridge of carbon atoms with Ar or  $R^3$  or  $R^4$  possibly comprising heteroatoms

$R^3$ ,  $R^4$  which can be equal or different, each independently represent hydrogen, linear or branched  $C_1$ - $C_{12}$  alkyl, phenyl, chlorine, bromine,  $-SO_3H$  possibly salified with organic or inorganic bases, or  $ArCO$ -

Ar represents a  $C_6$ - $C_{10}$  aryl either non-substituted or carrying one or more substituents of the halogen,  $C_1$ - $C_{12}$  alkyl, phenyl,  $C_1$ - $C_4$  alkoxy,  $C_3$ - $C_6$  cycloalkoxy, phenoxy,  $-COOH$ ,  $C_1$ - $C_6$  alkoxycarbonyl, benzoyl,  $C_1$ - $C_6$  dialkylamino,  $C_1$ - $C_6$  alkylthio, alkylsulphinyl, alkylsulphonyl, arylthio, arylsulphinyl and arylsulphonyl group; or 2,3 or 4-pyridyl; or a furan or thiophene residue

B) an organic tertiary amine of aliphatic type, or an ester of p-dimethylaminobenzoic acid or 4,4'-bis-( $C_1$ - $C_6$  dialkylamino) benzophenone, or single or polymeric ethers of primary or secondary alcohols or glycols or primary or secondary

aliphatic alcohols.

2. The use in accordance with claim 1 of systems consisting of:

- 5 A) one or more benzophenones of formula I in which  
R, R<sup>1</sup>, R<sup>2</sup> which can be equal or different, each independently  
signify C<sub>1</sub>-C<sub>4</sub> alkyl,  
R<sup>3</sup>, R<sup>4</sup> which can be equal or different, each independently  
signify hydrogen, C<sub>1</sub>-C<sub>4</sub> alkyl, or -SO<sub>3</sub>H possibly  
10 salified with organic or inorganic bases,  
Ar signifies phenyl, possibly substituted with one or  
more C<sub>1</sub>-C<sub>4</sub> alkyl, Cl, Br or C<sub>1</sub>-C<sub>4</sub> alkylthio groups  
B) an organic tertiary amine of aliphatic type, or an ester  
of p-dimethylaminobenzoic acid or 4,4'-bis-(C<sub>1</sub>-C<sub>4</sub> dialkylamino)  
15 benzophenone, or molecules containing single or polymeric  
derivatives of glycols.

3. The use in accordance with claims 1 and 2 of systems consisting of:

- 20 A) one or more benzophenones of formula I chosen from 2,4,6-trimethylbenzophenone; 2,4,6-triethylbenzophenone; 2,4,6-triisopropylbenzophenone; 2,3,4,5,6-pentamethylbenzophenone  
B) tertiary amines, or alcohols or ethers as defined in  
claim 1.

25

4. The use in accordance with claims 1, 2 and 3 of systems in which component A consists of one or more compounds of formula I, preferably chosen from 2,4,6-trimethylbenzophenone, 2,4,6-triethylbenzophenone, 2,4,6-triisopropylbenzophenone and  
30 2,3,4,5,6-pentamethylbenzophenone, in mixture with benzophenone, said mixture having a melting point considerably lower than that of the individual components.

5. The use in accordance with claims 1, 2, 3 and 4 of  
35 systems in which component A consists of a mixture of 2,4,6-trimethylbenzophenone and benzophenone in a molecular ratio of between 10:1 and 1:10 and preferably between 1.3:1 and 1:1, said

mixture being liquid above 10°C.

6. A photopolymerisable system consisting of:
- a) ethylenically unsaturated monomers and/or oligomers preferably of acrylic or methacrylic type,
  - b) mixtures of benzophenones and tertiary amines as photochemical polymerisation initiators defined as in claims 1 to 5,
  - c) other compounds such as pigments, fillers, dyes, stabilisers and various auxiliaries.
7. A mixture consisting of 2,4,6-trimethylbenzophenone and benzophenone in a molecular ratio of between 1.3:1 and 1:1, having the characteristic of being liquid at temperatures exceeding 10°C.

(12)

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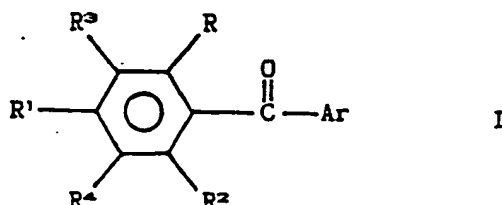
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in which R, R¹ and R² are preferably alkyls of 1-4 carbon atoms, R³ and R⁴ are preferably hydrogen and Ar is preferably phenyl, in combination with donors of hydrogen or substances which determine the formation of labile photochemical complexes, such as alcohols, ethers and tertiary amines with hydrogens in the alpha position, and the use for the same purpose of mixtures of 2,4,6-trialkylbenzophenones and benzophenone and particularly of a mixture of 2,4,6-trimethylbenzophenone and benzophenone in a molar ratio of 1.155, which has the advantage of being liquid at temperatures exceeding 10°C and of possessing photochemical activity superior to that of benzophenone.

EP 0 209 831 A3

European Patent  
Office

## EUROPEAN SEARCH REPORT

0209831

Application number

EP 86 10 9669

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)
A	US-A-4 071 424 (E.C. DART et al.)		C 08 F 2/50 G 03 C 1/58
A	US-A-4 080 275 (J.A. PHOTIS et al.)		
A	US-A-3 686 084 (H.J. ROSENKRANZ et al.)		
			TECHNICAL FIELDS SEARCHED (Int. Cl. 4)
			C 08 F G 03 C
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 22-01-1987	Examiner CAUWENBERG C.L.M.
CATEGORY OF CITED DOCUMENTS			
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